

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

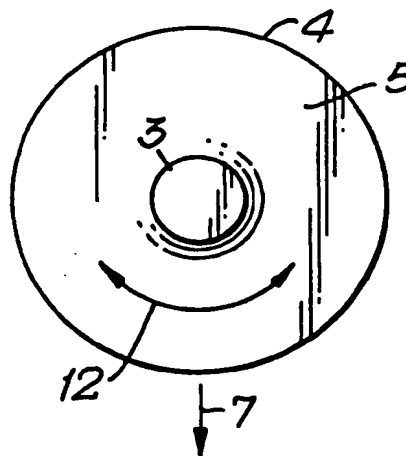
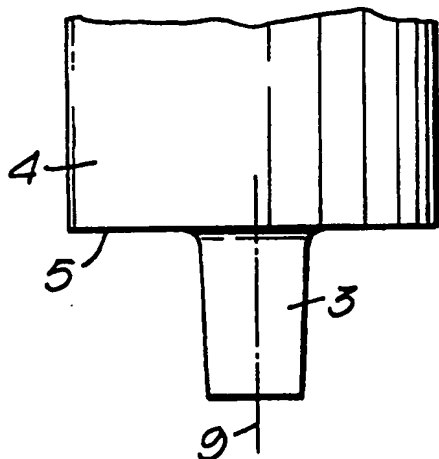
**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : B23K 20/12, B29C 65/06	A1	(11) International Publication Number: WO 96/38256 (43) International Publication Date: 5 December 1996 (05.12.96)
(21) International Application Number: PCT/GB96/01285 (22) International Filing Date: 29 May 1996 (29.05.96) (30) Priority Data: 9510859.3 30 May 1995 (30.05.95) GB (71) Applicant (for all designated States except US): THE WELD- ING INSTITUTE [GB/GB]; Abington Hall, Abington, Cam- bridge CB1 6AL (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): NICHOLAS, Edward, David [GB/GB]; 106 Abbots Road, Haverhill, Suffolk CB9 ODH (GB). THOMAS, Wayne, Morris [GB/GB]; 6 Howe Road, Haverhill, Suffolk CB9 9NJ (GB). NEEDHAM, James, Christopher [GB/GB]; 5 Blacklands Close, Saffron Walden, Essex CB11 4BX (GB). (74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).		(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>

(54) Title: IMPROVEMENTS RELATING TO FRICTION WELDING



(57) Abstract

A method of joining workpieces defining a joint region therebetween. The method comprises carrying out the following steps without causing relative bodily movement between the workpieces: 1) cause a probe (3) of material harder than the workpiece material to enter the joint region and opposed portions of the workpieces on either side of the joint region while causing relative cyclic movement between the probe (3) and the workpieces whereby frictional heat is generated to cause the opposed portions to take up a plasticised condition; 2) remove the probes (3); 3) allow the plasticised portions to solidify and join the workpieces together. The relative cyclic movement comprises repeatedly causing relative movement between the probe (3) and the workpieces in one direction and then in the opposite direction about an axis extending through the joint region between the workpieces.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

IMPROVEMENTS RELATING TO FRICTION WELDING

The invention relates to methods of joining workpieces defining a joint region therebetween and methods of
5 operating on a workpiece. The invention also relates to probes for use in these methods.

In EP-A-0615480 we introduced a concept known as friction stir welding which is a method of joining workpieces defining a joint region therebetween comprising
10 carrying out the following steps without causing relative bodily movement between the workpieces: causing a probe of material harder than the workpiece material to enter the joint region and opposed portions of the workpieces on either side of the joint region while causing relative
15 cyclic movement between the probe and the workpieces whereby frictional heat is generated to cause the opposed portions to take up a plasticised condition; removing the probe; and allowing the plasticised portions to solidify and join the workpieces together.

20 In the friction stir process, the tool or probe in the past has been either rotated, or oscillated (vertically) in the through thickness plane of the joint faces. Both methods have advantages and disadvantages. For example, with a rotating probe the mutual joint line or track seam
25 can be substantially straight or curved, or even change direction through any angle including forming a seam at right angles, and so forth. However, with the rotating probe, there is a degree of asymmetry between the "advancing" and "retreating" sides of the probe where the
30 motion is in the same direction as, or contrary to, the direction of travel. This asymmetry can lead to the formation of a continuous void on one side in the joint zone.

On the other hand, with the reciprocating probe blade,
35 the plasticised layer forms equally on either side on the blade. The joint line has to be substantially straight with the narrow blade in line with the joint. For curved

joint lines, especially with a relatively small radius of curvature, the blade is preferably curved with substantially the same radius of curvature. Moreover, with a narrow blade, if the blade is not accurately aligned with the joint, then the plasticised zones are formed in one material alone, and a joint between two components is not achieved.

In the same European patent specification, we introduced the concept of friction plunge welding defined as a method of operating on a workpiece comprising offering a probe of material harder than the workpiece material to a continuous or substantially continuous surface of the workpiece, the probe depending from a containment member having a surface which faces the workpiece; causing relative cyclic movement between the probe and the workpiece while urging the probe and workpiece together whereby frictional heat is generated as the probe enters the workpiece so as to create a plasticised region in the workpiece material around the probe, the containment member substantially preventing dispersal of the plasticised material; stopping the relative cyclic movement; and allowing the plasticised material to solidify around the probe.

This process is difficult to carry out with large probes.

In accordance with one aspect of the present invention, a method of joining workpieces defining a joint region therebetween comprises carrying out the following steps without causing relative bodily movement between the workpieces: causing a probe of material harder than the workpiece material to enter the joint region and opposed portions of the workpieces on either side of the joint region while causing relative cyclic movement between the probe and the workpieces whereby frictional heat is generated to cause the opposed portions to take up a plasticised condition; removing the probe; and allowing the plasticised portions to solidify and join the workpieces

together and is characterised in that the relative cyclic movement comprises repeatedly causing relative movement between the probe and the workpieces in one direction and then in the opposite direction about an axis extending
5 through the joint region between the workpieces.

In accordance with a second aspect of the present invention, a method of operating on a workpiece comprises operating on a workpiece, the method comprising offering a probe of material harder than the workpiece material to a
10 continuous or substantially continuous surface of the workpiece, the probe depending from a containment member having a surface which faces the workpiece; causing relative cyclic movement between the probe and the workpiece while urging the probe and workpiece together
15 whereby frictional heat is generated as the probe enters the workpiece so as to create a plasticised region in the workpiece material around the probe, the containment member substantially preventing dispersal of the plasticised material; stopping the relative cyclic movement; and
20 allowing the plasticised material to solidify around the probe and is characterised in that the relative cyclic movement comprises repeatedly causing relative movement between the probe and the workpieces in one direction and then in the opposite direction about an axis extending
25 through the joint region between the workpieces.

In this invention, we propose that the probe is oscillated about an axis extending through the joint region between the workpieces and this has the advantage that the plasticised material is formed substantially symmetrically
30 on either side of the probe body, and can be used on both straight and curved joint lines. As will be appreciated, this arrangement overcomes the disadvantages of the two prior art methods.

Typically, the relative cyclic movement will comprise
35 a rotation and in some examples the cyclic movement could comprise a limited number of full revolutions in one direction followed by a similar number of revolutions in

the other direction. For example, up to ten or twenty revolutions in each direction. Although the motion is continuous in any one direction for a limited period, this does not lead to a build up of the cavity or void which has been experienced before. However, preferably, the degree of movement in each direction is less than a full rotation of the probe about the axis.

In some cases, the axis about which the cyclic movement occurs is displaced from the probe so as to define a bodily arcuate or orbital movement. Preferably, however, the probe has an elongate axis which is coincident with the said axis about which the relative cyclic movement is caused.

In accordance with a third aspect of the present invention, we provide a probe for use in a method of joining workpieces defining a joint region therebetween, the probe having an aperture extending therethrough.

In accordance with a fourth aspect of the present invention, we provide a probe for use in a method of operating on a workpiece, the probe having an aperture extending therethrough.

The methods with which the probes can be used may include methods of the type described in EP-A-0615480 and methods in accordance with the first and second aspects of the present invention.

The presence of an aperture extending transversely through the probe is advantageous for some material where the joint surfaces tend to remain unbonded in spite of being heated to a degree of softening. For plastic material, particularly some thermoplastics, the surfaces of the material can remain with a lower degree of bond strength in spite of being heated and pressed together. This surface effect is avoided by the probe as described where the central region of the probe allows the break up of the joint surface as it passes through.

In addition, or alternatively, the aperture may extend axially through the probe.

In some examples, the aperture communicates with means for supplying material to the joint region via the probe. This is particularly advantageous with plastics materials such as thermoplastics, since it allows pre-heated material to be extruded through the probe to supply additional plasticised material to the joint region. This is beneficial especially where there is a poor fit between the components to be joined, or to allow a degree of thickening in the joint region.

Some examples of methods and probes according to the invention will now be described and contrasted with known methods with reference to the accompanying drawings, in which:-

Figures 1 and 2 illustrate known methods of friction stir welding;

Figure 3 is a side elevation of a probe and support;

Figures 4 and 5 are end elevations of two alternative probes;

Figures 6-8 are similar to Figures 3-5 but illustrating probes with laterally extending apertures; and,

Figures 9-11 are longitudinal sections through three further probe constructions each having an axially extending aperture.

The example shown in Figure 1 is described in more detail in EP-A-0615480. Briefly, a pair of aluminium alloy plates 1A,1B are butted together about a joint line 2. A non-consumable probe 3 of steel supported beneath a cylindrical member 4 defining a shoulder 5 is brought to the edge of the joint line 2. The probe 3 is rotated by a motor 6 while the probe is traversed in the direction 7 and while the plates are held against lateral movement away from the probe 3. The rotating probe 3 produces a local region of highly plasticised material in each workpiece 1A,1B and following passage of the probe, this plasticised material is allowed to solidify thereby joining the

workpieces together. The shoulder 5 acts to contain the plasticised material.

In a second example of the prior art, shown in Figure 2, the probe 3 is replaced by a reciprocating blade 8 which is traversed along the joint line 2.

In these two examples, the probe 3 or the blade 8 is removed following its action. In another example (not shown but which again is described in EP-A-0615480) the probe is allowed to remain in situ (friction plunge welding).

It will be appreciated that using a rotating probe, there is a tendency to take material from the lefthand side and deposit it towards the righthand side as shown in Figure 1. This tendency can result in a void being formed on the lefthand side which is present throughout the seam. The same effect but to a lesser extent can occur in friction plunge welding. To eliminate this effect, a suitable close fit between the body of the probe and the materials to be joined is usually required. This problem does not occur with the method described in Figure 2 but then, as the blade 8 is relatively extended in the direction of travel and is relatively narrow, it must be accurately aligned with the joint seam and the joint seam must be substantially straight. (In an alternative arrangement for curved joint lines, the blade may be giving a similar curved shape so as to lie within the joint seam as it is being made.)

In the preferred example of the present invention, the probe 3 is rotated to and fro in an arcuate manner about its axis 9 as it traverses 7 along the joint line 2 (Figure 3). As will be appreciated, this motion is symmetrical with respect to either side of the joint line, and the problem of asymmetric movement of the plasticised material is avoided.

Typically, the probe 3 will have a circular cross-section (Figure 4) and this has a further advantage over

the blade arrangement of Figure 2 in that it can traverse the joint line in any direction.

In an alternative form, the probe 3' may have a semicircular portion 10 facing the direction of travel 7 and straight edge portions 11 at the rear (Figure 5).

In these examples, arrows 12 illustrate the degree of motion in each direction. In other examples (not shown) the probe could rotate further and could even fully rotate one or more times and then be reversed to rotate in the other direction by the same amount. For this, conveniently two drives operable in opposite directions can be used, each one being coupled in turn to the probe.

In the Figure 3 example, the shoulder 5 presents a plane orthogonal to the probe 3. In some cases, it is preferable to angle the plane of the shoulder relative to the probe 3.

Preferably, the arcuate motion of the probe is substantially symmetrical about the tangent to the joint line at the position of the probe.

Figures 6-8 illustrate a variation of the probes shown in Figures 3-5. In these examples, each probe 3'', 3''' has an aperture 13 extending through it generally in line with the direction of traverse 7. The advantages of the aperture 13 have been mentioned above.

In a further alternative, the probe is constructed with a central axial hollow region or bore 14 (Figures 9-11) which opens into an enlarged bore 15 in the cylindrical portion 4. This bore 15 can be partially filled with an additional heated material such as aluminium which is passed into the joint 2. This further material can be substantially similar to the parent materials being joined or alternatively can be a different material to improve the properties of the joints so formed. The material is urged towards the bore 14 with a plunger 17 and a set of spring washers 18 (Figures 9 and 11) or an auger 18 (Figure 10). The spring washers 18 act against a cap 19 secured to the cylinder 4.

In this case, the cylindrical portion 4 is surrounded by an induction coil 20 and a ferrite magnetic flux intensifier 21.

Although the bore 14 has been shown as extending axially, in other arrangements (not shown) the probe may have one or more apertures to its rear so that the additional filler material passes into the joint at the rear, or the or each aperture could be provided facing forward. In further examples, both forward and rearward opening apertures could be provided or a series of small apertures could be positioned circumferentially around the probe.

The apparatus for generating the arcuate motion of the probe 3 could comprise a simple link mechanism. In this, one end of the link is attached to the probe (or cylindrical portion 4) and the other end is attached to a boss (not shown) which is not concentric with the rotating cylindrical portion 4. The eccentricity causes the probe 3 to be moved in the arcuate manner described as the probe is constrained to remain coaxial with its support. In an alternative arrangement, an internal cam arrangement could be provided which causes an arcuate output motion from a rotating input shaft. These and other mechanisms can be used as appropriate giving rise to rotational arcuate motion with a frequency of at least 10 Hz and preferably in excess of 50 Hz.

As an alternative to the mechanical arrangements, the probe could be oscillated in an arcuate manner using electromechanical or hydraulic techniques.

A further advantage can be achieved by tilting the probe, typically in the range 1-3° to the vertical, to ensure that the forward angle between the axis of the probe and the surface of the workpiece in the direction of travel is not less than 90° (normal) but preferably slightly greater. In addition, we believe that a tilt transverse to the direction of travel is also advantageous in certain circumstances.

The friction stir welding process has particular advantages when carried out under water. Firstly, the process can be used to weld components under water in, for example, sub-sea and ship repair situations but it may also
5 provide certain metallurgical advantages. For example, increased cooling rate will lead to a reduced heat affected zone and assist improvement in the chemical properties with some materials.

CLAIMS

1. A method of joining workpieces defining a joint region therebetween, the method comprising carrying out the following steps without causing relative bodily movement between the workpieces: causing a probe of material harder than the workpiece material to enter the joint region and opposed portions of the workpieces on either side of the joint region while causing relative cyclic movement between the probe and the workpieces whereby frictional heat is generated to cause the opposed portions to take up a plasticised condition; removing the probe; and allowing the plasticised portions to solidify and join the workpieces together, characterised in that the relative cyclic movement comprises repeatedly causing relative movement between the probe and the workpieces in one direction and then in the opposite direction about an axis extending through the joint region between the workpieces.
2. A method according to claim 1, wherein the joint region has an elongate dimension extending laterally between the workpieces, the method further comprising causing relative translational movement between the workpieces and the probe in the direction of the joint region.
3. A method according to claim 1 or claim 2, wherein the probe extends through the thickness of the workpieces.
4. A method according to any of claims 1 to 3, wherein the workpieces comprise separate members.
5. A method of operating on a workpiece, the method comprising offering a probe of material harder than the workpiece material to a continuous or substantially continuous surface of the workpiece, the probe depending from a containment member having a surface which faces the workpiece; causing relative cyclic movement between the probe and the workpiece while urging the probe and workpiece together whereby frictional heat is generated as the probe enters the workpiece so as to create a

plasticised region in the workpiece material around the probe, the containment member substantially preventing dispersal of the plasticised material; stopping the relative cyclic movement; and allowing the plasticised material to solidify around the probe, characterised in that the relative cyclic movement comprises repeatedly causing relative movement between the probe and the workpieces in one direction and then in the opposite direction about an axis extending through the joint region between the workpieces.

6. A method according to any of the preceding claims, wherein the relative cyclic movement comprises a rotation.

7. A method according to claim 6, wherein the degree of movement in each direction is less than a full rotation of the probe about the axis.

8. A method according to any of the preceding claims, wherein the probe has an elongate axis which is coincident with the said axis about which the relative cyclic movement is caused.

9. A probe for use in a method of joining workpieces defining a joint region therebetween, in which the method comprises carrying out the following steps without causing relative bodily movement between the workpieces: causing a probe of material harder than the workpiece material to enter the joint region and opposed portions of the workpieces on either side of the joint region while causing relative cyclic movement between the probe and the workpieces whereby frictional heat is generated to cause the opposed portions to take up a plasticised condition; removing the probe; and allowing the plasticised portions to solidify and join the workpieces together, the probe having an aperture extending therethrough.

10. A probe according to claim 9, wherein the aperture extends laterally across the probe to enable plasticised material to flow through the probe in use.

11. A probe according to claim 9, wherein the aperture communicates with means for supplying material to the joint region via the probe.
12. A probe for use in a method of operating on a
5 workpiece, the method comprising offering a probe of material harder than the workpiece material to a continuous or substantially continuous surface of the workpiece, the probe depending from a containment member having a surface which faces the workpiece; causing relative cyclic movement
10 between the probe and the workpiece while urging the probe and workpiece together whereby frictional heat is generated as the probe enters the workpiece so as to create a plasticised region in the workpiece material around the probe, the containment member substantially preventing
15 dispersal of the plasticised material; stopping the relative cyclic movement; and allowing the plasticised material to solidify around the probe, the probe having an aperture which extends through the probe to enable material to be supplied to the joint region via the probe.
- 20 13. A probe according to any of claims 9 to 12, further comprising means within the probe for urging material out through the aperture.
14. A probe according to claim 13, wherein the means includes a compression spring.
- 25 15. A probe according to claim 13, wherein the means includes an auger.
16. A probe according to any of claims 9 to 15, depending from a containment shoulder of a support member, the containment shoulder being planar and oriented in a non-
30 orthogonal manner relative to the probe.
17. A method according to any of claims 1 to 8, wherein the probe is constructed in accordance with any of claims 9 to 16.

$\frac{1}{3}$

Fig.1.

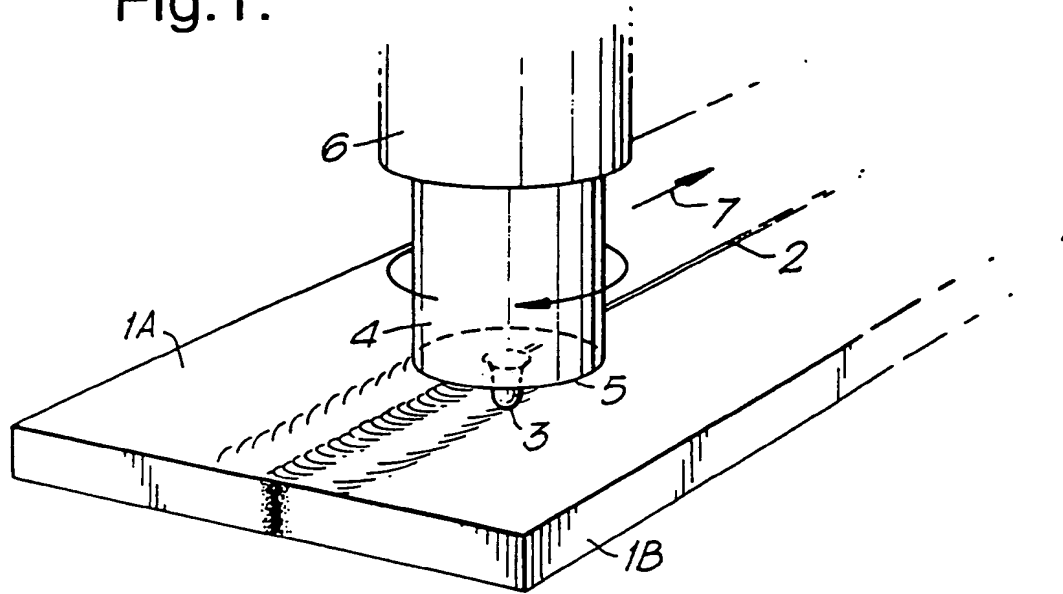
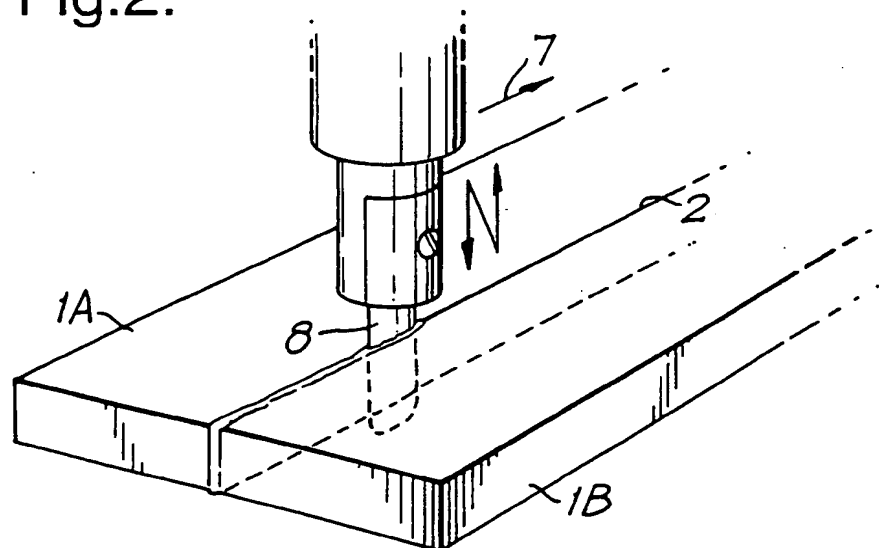


Fig.2.



SUBSTITUTE SHEET (RULE 26)

2/3

Fig.3.

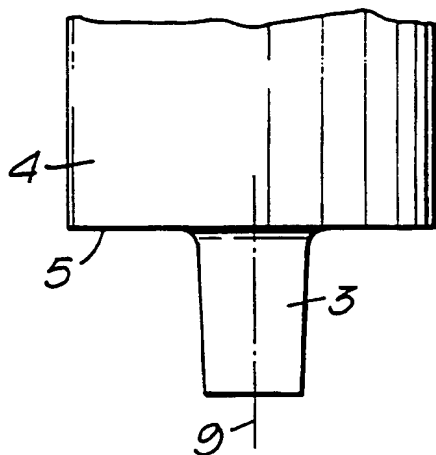


Fig.4.

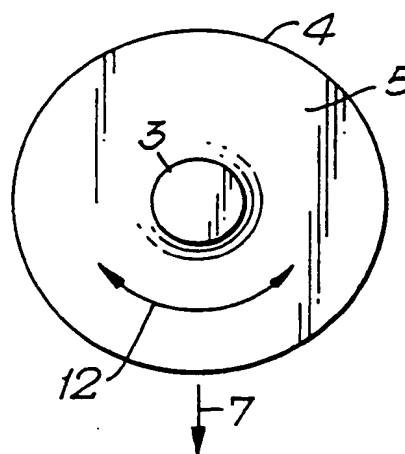


Fig.5.

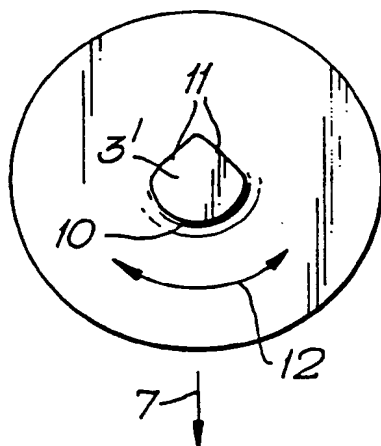


Fig.6.

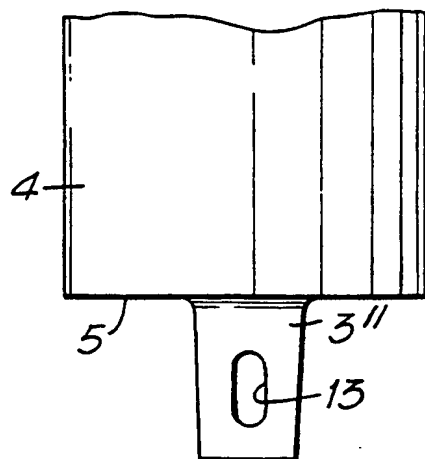


Fig.7.

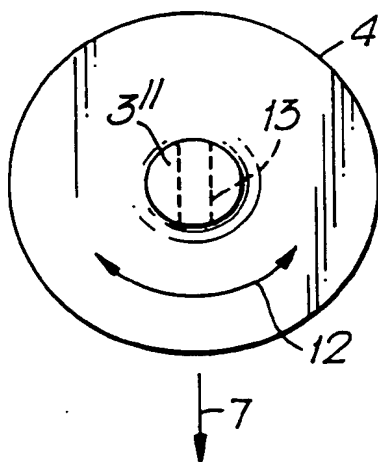
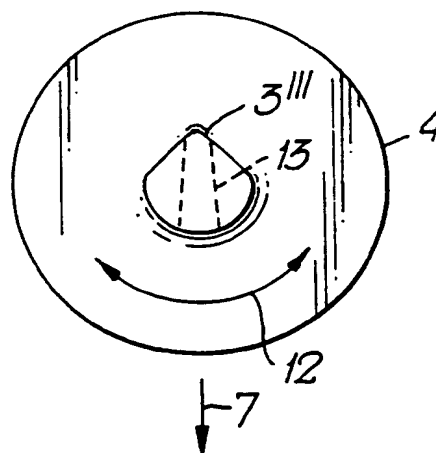


Fig.8.



SUBSTITUTE SHEET (RULE 26)

3/3

Fig.9.

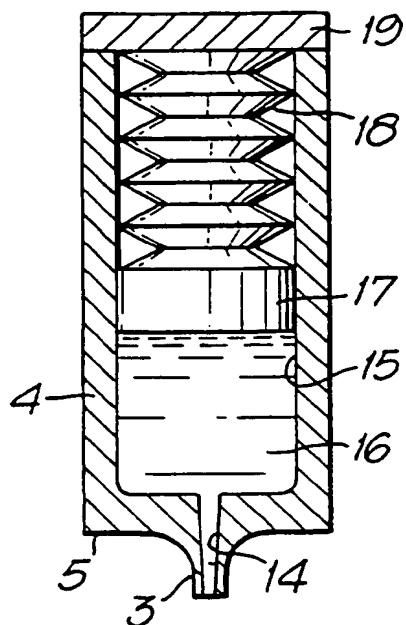


Fig.11.

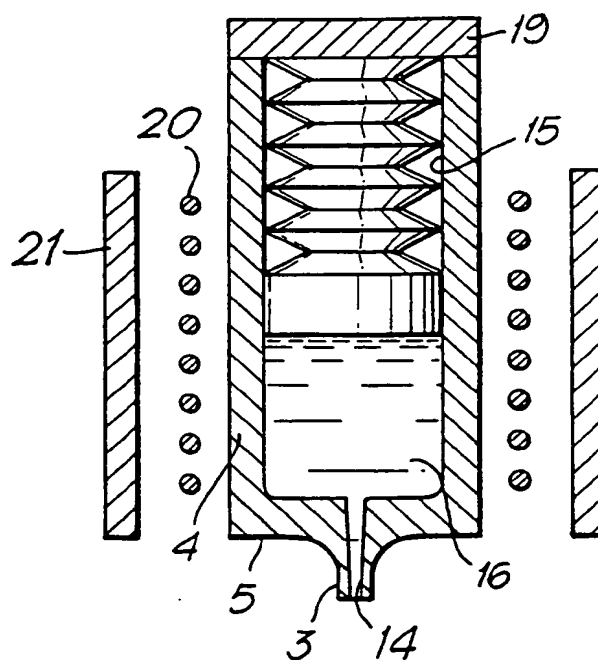
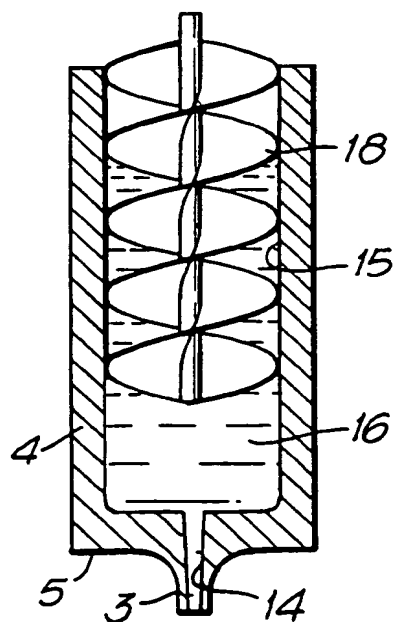


Fig.10.



SUBSTITUTE SHEET (RULE 26)

INTERNATIONAL SEARCH REPORT

Internat. Application No
PCT/GB 96/01285A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 B23K20/12 B29C65/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 B23K B29C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO,A,93 10935 (WELDING INST) 10 June 1993 see page 2, line 1 - page 4, line 4; figures 1,12	1-6,9, 12,17
A	& EP,A,0 615 480 cited in the application ---	
A	US,A,4 405 851 (SHELDON GARY L) 20 September 1983 see column 9, line 49 - line 56; figures 4,5 ---	1,5-9, 12,17
A	GB,A,2 270 864 (WELDING INST) 30 March 1994 see page 9, line 29 - page 10, line 22; figures 4-6 ---	1,5, 8-12,17

	-/--	

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *G* document member of the same patent family

Date of the actual completion of the international search

2 September 1996

Date of mailing of the international search report

13.09.96

Name and mailing address of the ISA
European Patent Office, P.B. 5818 Patentaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+31-70) 340-3016

Authorized officer

Rausch, R

INTERNATIONAL SEARCH REPORT

Intern: I Application No
PCT/GB 96/01285

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 337 691 (WELDING INST) 18 October 1989 ---	
A	DE,A,41 03 740 (BRANSON ULTRASCHALL) 13 August 1992 ---	
A	DE,A,42 06 583 (FRAUNHOFER GES FORSCHUNG) 9 September 1993 ---	
A	WO,A,94 25214 (ADVANCED SURFACES AND PROCESSE) 10 November 1994 -----	

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internat Application No

PCT/GB 96/01285

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A-9310935	10-06-93	AU-B- 662310	31-08-95
		AU-B- 1016495	30-03-95
		AU-B- 658950	04-05-95
		AU-B- 2952892	28-06-93
		CA-A- 2123097	10-06-93
		DE-D- 69205991	14-12-95
		DE-T- 69205991	11-04-96
		EP-A- 0615480	21-09-94
		EP-A- 0653265	17-05-95
		JP-T- 7505090	08-06-95
		US-A- 5460317	24-10-95
US-A-4405851	20-09-83	NONE	
GB-A-2270864	30-03-94	NONE	
EP-A-0337691	18-10-89	DE-D- 68912633	10-03-94
		JP-A- 1306571	11-12-89
		US-A- 4959241	25-09-90
DE-A-4103740	13-08-92	NONE	
DE-A-4206583	09-09-93	NONE	
WO-A-9425214	10-11-94	US-A- 5448035	05-09-95
		AU-B- 6669494	21-11-94